IS 327 (1991): Oil of Lemongrass [PCD 18: Natural and Synthetic Fragrance Materials]

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IS 327 : 1991
(Reaffirmed 2002)

भारतीय मानक

लेमनग्रास तेल — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

OIL OF LEMONGRASS — SPECIFICATION

(Second Revision)

UDC 665·524·26

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1991

Price Group 2

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1952 and first revised in 1961. The Sectional Committee responsible for its preparation felt that the standard should be revised with a view to bring it in line with trade practices prevalent in perfumery industry and also to align the quality level of material currently being manufactured and sold in the country.

In the earlier revision, determination of citral content using freshly prepared 30 percent *m/v* solution of sodium bisulphite was prescribed. In this revision hydroxylamine method is being included as an alternative method for determination of citral content. Gas Layer Chromatographic method for determination of neral and geranial content is being included for guidance only under Annex B.

In India, oil of lemongrass is produced in the South-Western State of Kerala by the distillation of lemongrass (*Cymbopogon flexuosus* Stapf fam. Gramineae). This essential oil produced largely in India since ages has come to be known as the East Indian Oil of Lemongrass and has found favour in the world market particularly because of its uniformly high citral content and the solubility in 70 percent alcohol of most of the oil produced in the country. This solubility in alcohol is regarded as synonymous with freshness and freedom from adulterants; though investigations have disclosed that some specimens of freshly distilled oil are also found to be insoluble in alcohol, if there is adventitious mixture, even in small proportions, with a similar but botanically different white-stemmed grass, locally known as '*wella poolu*' found in the fields. The white-stemmed grass now identified as *Cymbopogon flexuosus* Stapf Var. *albescens* has, however, been almost rooted out of the lemongrass fields by intensive efforts of the State Departments of Agriculture and Forests. The solubility of all specimens of the oil is known to be reduced on storage.

The oil produced in other countries, most notably West Indies, is insoluble in 70 percent alcohol. The lower solubility of the 'West Indian' oil, particularly noticeable after storage of the freshly distilled oil, is due to the presence, in the foreruns, of myrcene, an olefinic terpene, which on exposure to air and light readily polymerizes. This difference in the quality of the oil is attributed to the different plant species, *Cymbopogon citratus* Stapf. (fam. Gramineae) from which the 'West Indian' oil is derived.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified values in this standard.

Indian Standard

OIL OF LEMONGRASS — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of test for the material commercially known as the oil of lemongrass or the East Indian Oil of lemongrass. The material is largely used in the extraction of citral, the chief constituent of the oil and the starting material for the manufacture of important ionones. It is also employed in the preparation of lemon-like perfumes, germicides, etc.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 1980	Relative density (*second revision*)
(Part 4) : 1980	Determination of optical rotation (*second revision*)
(Part 5) : 1986	Determination of refractive index (*second revision*)
(Part 6) : 1986	Determination of solubility in ethanol (*second revision*)
(Part 11) : 1986	Determination of carbonyl value and content of carbonyl compounds (*second revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (*first revision*)

3 TERMINOLOGY

For the purpose of this standard, the definitions of terms given in IS 6597 : 1988 shall apply.

4 SAMPLING

Representative samples of the material, shall be drawn as prescribed in IS 326 (Part 1) : 1984.

5 REQUIREMENTS

5.1 Description

Oil of lemongrass shall be obtained by water or steam distillation of the freshly cut and partially dried grass botanically known as *Cymbopogon flexuosus*, Stapf fam. Gramineae.

5.1.1 Oil of lemongrass shall be a clear liquid, free from sediment, suspended matter, separated water and added adulterants.

5.1.2 The oil shall be examined for its colour, clarity, separated water, by notes and sediment, as prescribed under IS 326 (Part 2) : 1980.

5.2 Solubility

Oil of lemongrass shall be soluble in 3 volumes of ethyl alcohol (70 percent by volume), occasionally with slight turbidity, when tested as prescribed in IS 326 (Part 6) : 1986.

5.3 Oil of lemongrass shall also comply with the requirements given in Table 1.

Table 1 Requirement for Oil of Lemongrass

Sl No.	Characteristic	Requirement	Method of Test, Ref to IS No.
(1)	(2)	(3)	(4)
i)	Colour and appearance	Dark yellow to light brown-red mobile liquid	IS 326 (Part 2) : 1980
ii)	Odour	Lemon-like	IS 2284 : 1988
iii)	Relative density*, 27°/27°C	0·886 to 0·896	IS 326 (Part 3) : 1980
iv)	Optical rotation	− 3° to + 1°	IS 326 (Part 4) : 1980
v)	Refractive index†, 27°C	1·477 4 to 1·483 4	IS 326 (Part 5) : 1986
vi)	Citral content, percent by volume, *Min*	75	Annex A

*The correction factor for relative density for each degree Celsius change in temperature is 0·000 62.

†The corection factor for refractive index for each degree Celsius change in temperature is 0·000 44.

6 PACKING AND MARKING

6.1 Packing

The material shall be supplied in air-tight containers preferably glass, tin lined or aluminium; or as agreed to between the purchaser and the supplier.

6.1.1 The material shall be protected from light and stored in cool and dry place.

6.2 Marking

The material shall be marked with the following information:

a) Name of the material,
b) Indication of the source of manufacture,
c) Net mass of the material and percentage of total citral content, and
d) Batch number.

6.2.1 The containers may also be marked with the Standard Mark.

7 SAMPLING

Representative samples of the material, shall be drawn as prescribed in IS 326 (Part 1) : 1984.

8 TESTS

8.1 Tests shall be carried out as prescribed under **5.1, 5.2** and the appropriate references specified in col 4 of Table 1.

8.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF CITRAL CONTENT

A-0 GENERAL

A-0.1 Two methods, namely, hydroxylamine method and sodium bisulphite methods are being prescribed for determining citral content of oil of lemongrass. Any one of these may be adopted for determining citral content or as may be agreed to between the purchaser and the supplier. The procedure for hydroxylamine method as given under IS 326 (Part 11) : 1986 may be followed. Sodium bisulphite method has been prescribed under this Annex.

NOTE — It is of utmost importance always to record the method used when reporting an analytical result.

A-1 REAGENTS

A-1.1 Sodium Bisulphite Solution

Freshly prepared, 35 percent (m/v).

A-2 PROCEDURE

A-2.1 Introduce 75 ml of the sodium bisulphite solution measured from a graduated cylinder, into a cassia flask (Fig. 1). Pipette exactly 10 ml of the material into the flask and shake thoroughly. Immerse the flask in a boiling water-bath and shake repeatedly, until the solid addition compound goes completely into solution. Make a further addition of 25 ml of the bisulphite solution and again shake the flask repeatedly for one-half to one hour to ensure complete reaction of the carbonyl compound with the bisulphite solution. After allowing the cassia flask to stand undisturbed in boiling water for 10 minutes to permit the unreacted material to rise to the surface, add sufficient volume of sodium bisulphite solution to raise the residual material into the neck of the flask. Gently tap the flask and rotate it rapidly between the plams of the hands to raise droplets of materials adhering to the sides of the flask into the neck. After cooling the flask to room temperature, measure the volume of the residual material.

A small amount of the bisulphite addition compound often precipitates out of solution, sometimes at the surface when the material and aqueous layers meet, thus rendering exact reading difficult. If so, add a few drops of water in such a way that the water runs down along the inside of the neck of the flask so that it may remain temporarily on top of the bisulphite solution and give a sharp separation of the material and aqueous layers.

NOTE — If the material contains heavy metals, these should be removed before the determination by shaking the material thoroughly with a small amount (about 1 percent) of powdered tartaric acid and filtering, a sharper separation of the residual material will then result.

A-2.2 Calculation

Calculate the citral content by the following formula:

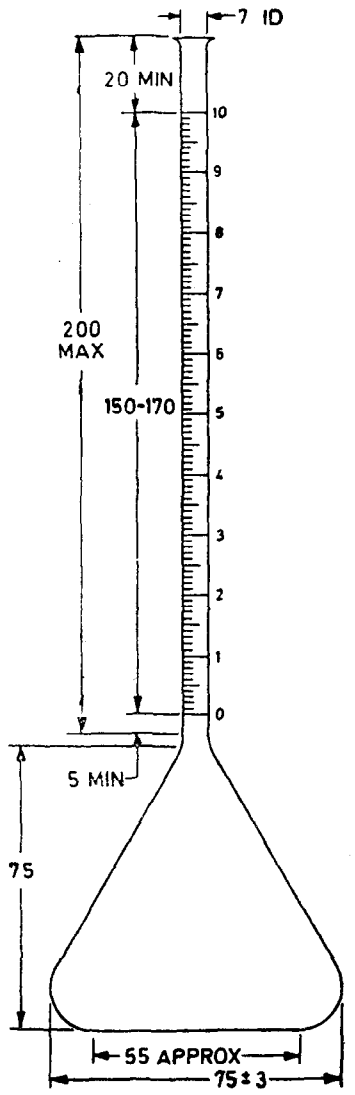
Citral content,
percent by volume $= 10 (10 - v)$

where

v = volume in ml of residual material.

NOTE — Citral content may be expressed as percent by mass (M) if the relative density of the original material and of the carbonyl compound is known at the same temperature, using the formula:

$$M = v \times \frac{\text{Relative density of carbonyl compond}}{\text{Relative density of material}}$$



All dimensions in millimetres.

FIG. 1 CASSIA FLASK

ANNEX B

(*Clause* 0.2)

GAS CHROMATOGRAPHIC ANALYSIS FOR OIL OF LEMONGRASS

B-0 GENERAL

B-0.1 The chromatographic conditions given here are for guidance only.

B-0.2 Outline of the Method

A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and

petroleum ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

B-1 APPARATUS

B-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for oil of lemongrass using a chromatograph with the following chromatographic conditions is shown in Fig. 2.

Sample	*Oil of Lemongrass*
Column	AT — 1 000
Material	Stainless steel
Length	5 m
Orifice	0·32 cm
Stationary phase and solid support*	10 percent by mass on chromosorb WHP 100-120 mesh
Carrier Gas	Nitrogen
Conditions	
Column temperature	190°C
Injection port temperature	250°C
Detector	
Type	F.I.D
Temperature	250°C

*The analysis may also be accomplished with columns containing : DEGS (Diethylene glycol succinate) and FFAP (Free Fatty Acid Phase) in carbowax 20 M treated with nitrophthalic acid.

B-2 CALCULATION

B-2.1 Area Measurements (*see* Note 1)

Since normal peaks approximates a triangle, the area is measured by multiplying the peak height times the width of the half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

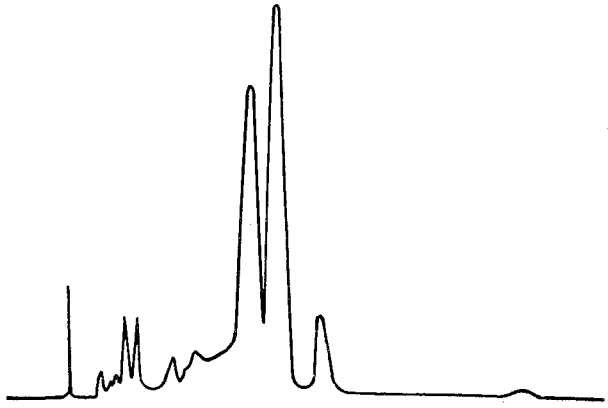


FIG. 2 OIL OF LEMONGRASS

B-2.2 Area Normalization (*see* Note 2)

By normalizing, it is meant calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTES

1 Other methods of area measurements, namely triangulation, disc integrator and electronic digital integrator if fixed with GLC machine would be of great advantage.

2 Internal standardization may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

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Indian Standards are reviewed periodically and revised, when necessary and amendments, if any, are issued from time to time. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition. Comments on this Indian Standard may be sent to BIS giving the following reference:

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IS 327 : 1991 OIL OF LEMONGRASS — SPECIFICATION

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[*Page* 1, *Table* 1, *Sl No.* (v), *col* 3] — Substitute '1.479 9 to 1.485 9' *for* '1.477 4 to 1.483 4'.

(PCD 18)

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